

# Activation of the Technological Combustion Process of Oxide Systems by Different Modifying Additives

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## Abstract

This paper describes the possibility of effect on the thermokinetic characteristics of the combustion process (SH-synthesis) with the help of modifying additives of inorganic and organic compounds containing bound water and ammoniac group. SH-synthesis was carried out by means of the furnace method under the conditions of self-ignition of the sample ( $\text{SiO}_2 + 37.5\% \text{Al}$ ) placed in the preheated furnace. The combustion thermograms have shown that the modifying quartz additives exert a significant effect on all combustion parameters including the induction period, combustion rate, maximum combustion temperature and the change of heat conditions at the after burning stage (postprocesses). The presence of a sufficient amount of bound water and ammonia group in the modifying additives positively effects the increase in the amount of the matrix phase (corundum) and aluminium nitride in synthesis products.

## Key words:

Quartz; Mechanochemistry; Modification; Carbon- and Nitrogenous Additives; Nanostructures

## Introduction

One of the effective methods to obtain ceramic materials different in their compositions and functional purposes is synthesis under the conditions of solid phase combustion (or SH-synthesis) proceeding due to exothermic reactions between initial chemical elements of the fuel mixture (charge). The main characteristic of SHS is combustion rate whose significance depends on the composition of the fuel mixture and its physical characteristics (the size of particles, their power capacitance). When reaching the maximum temperature, the process progresses to the stage of after-combustion which is determined in formation of the phase composition and, hence, the properties of synthesis products.

There are numerous methods to influence the kinetic and thermal characteristics of the process which include both the preliminary preparation of the initial material: milling, mechanical activation and other ones providing the increase in power capacitance of the mixture; and direct effect on the development of chemical reactions in the combustion process, in particular, by introducing different additives which contribute to the increase in the amount of heat or formation of a gaseous medium providing the transfer of initial fuel components to the reaction zone.

Mechanochemical treatment (MCT) of powders in the presence of different modifying additives is an effective method to change the structure and state of the treated material particles. Under the conditions of mechanical effect, one should take into account the whole spectrum of phenomena and processes accompanying the deformation and destruction of a solid body, including formation and accumulation of defects, bond splitting, local heat build-up in the impact zone, all of which are combined to change the energy state and reactivity of the material and, hence, the chemistry on the surface of particles, regularities and kinetics of the reactions providing formation of new surface layers.

Among a great number of mineral carriers, silicon dioxide (silica) is widely spread and its chemical modification with different compounds has been studied most extensively. Modification of silica occurs mainly with participation of deformed or split siloxane and silanol groups in the reaction. Under mechanical effect Si-O bond splits on the surface of crystal, this provides addition of OH-groups with formation of hydrogen bonds. It is stated that the number of bound OH-groups is equal to the doubled

number of split Si-O bonds. As well, apart from surface groups, internal groups of SiOH are formed due to diffusion of water molecules into the solid structure for the distance of up to 15 nm. The presence of hydroxyl groups, their state, location and arrangement plays an important role in the modification process of the surface of particles. The most widely spread reaction of modification is the reaction of interaction of SiO<sub>2</sub> surface with alcohols (esterification of silanol groups with grafting of alcohol radical and water molecule).

This paper describes the possibility of effect on the thermokinetic characteristics of the process providing the complete procedure of the reaction between the main components of the reaction mixture in the course of MCT using inorganic and organic compounds containing bound water and ammoniac group as modifying additives. In the presence of aminogroups in the system, in the process of heating in the oxygen containing medium, water and nitrogen taking part in synthesis are formed.

SH-synthesis was carried out at stoichiometric ratio of the components (SiO<sub>2</sub>+37.5% Al). This system, on the one hand, can be considered as a model one, in study of the combustion process depending on different factors. On the other hand, silicon oxide is a component of many charge mixtures in production of SHS-refractories and heat insulators. The main products of the reaction between silicon dioxide and aluminium are corundum, silicon and mullite which are components of many refractory materials. The most complete consumption of initial components is determined by preparation of the charge and the conditions of SH-synthesis procedure. Here, the preliminary mechanochemical treatment of silicon dioxide can be an effective method to influence the development of the combustion process and production of synthesis products.

### Materials and Methods of Investigation

The investigations were carried out using quartz sand of Kuskuduk deposit with the content of quartz 81.3%. Besides, it contains up to 18.7% of microcline K(Si<sub>3</sub>Al)O<sub>8</sub>, as well as different elements saturating the surface and particles dissolved in the bulk. According to the results of spectral analysis, it contains 0.1–1.0% of iron, magnesium, calcium, sodium. Aluminum of the brand PA4 was used as a reducing agent. Nitrogen containing compounds:

dicyandiamide – HN=C(NH<sub>2</sub>)NHCN, carbamide – (NH<sub>2</sub>)<sub>2</sub>CO, aqueous ammonia solution (i.e. ammonium hydroxide – NH<sub>4</sub>OH) as well as succinic acid – H<sub>2</sub>C<sub>4</sub>H<sub>4</sub>O<sub>4</sub>, the mixture succinic acid +aqueous solution of ammonia – H<sub>2</sub>C<sub>4</sub>H<sub>4</sub>O<sub>4</sub>+ NH<sub>4</sub>OH, polyvinyl alcohol – (C<sub>2</sub>H<sub>3</sub>OH)<sub>n</sub>, water containing silicic acid – H<sub>2</sub>SiO<sub>3</sub>·n(H<sub>2</sub>O) served as modifying additives.

Quartz sand was mechanochemically treated in the mill of a dynamic action with acceleration of 20g during 10 minutes with the ratio of the mass of powder to the mass of balls 1:2. During MCT, up to 5% of the modifying additive was introduced to the powder treated. SH-synthesis was performed by the furnace method under the conditions of self-ignition of the sample (SiO<sub>2</sub>+37.5%Al) placed into the preheated furnace. The temperature of furnace heating made up to 900°C. The samples with the diameter of 20 mm and height 20 mm were moulded on the laboratory press "Carver" with the pressure of 10 t.

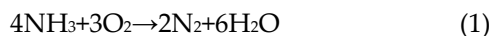
The combustion temperature was measured by a pyrometric thermometer IrconUltrimaxPlus UX 10P which is used for measuring temperature from 600 to 3000°C. The measurement error up to 1500°C makes up ± 0.5 % of the value measured and ±1% in the temperature range 1500 ÷ 2000°C. The time interval of registration of the device readings makes up 0.5s. We measured the temperature, the time before the sample ignition and the procedure time of the reaction itself and evaluated the rate of the reaction development to maximum temperature. The reaction completeness was determined by the phase composition of synthesis products. The morphology of the quartz powder particles modified by different additives was studied on the transmission electron microscope Jem -100CX; U-100kv. The objects to be investigated were prepared by the method of dry preparation. X-ray phase analysis of the synthesis products was performed on diffractometer DX-rayGP-4M using cobalt and copper K<sub>α</sub>-radiation in the range of 2θ= 10°-70°. When determining the phase contents, we performed a semi-quantitative analysis using the method of corundum numbers and integral intensity of diffraction lines of the compounds under study.

### Results of Investigation and Discussion

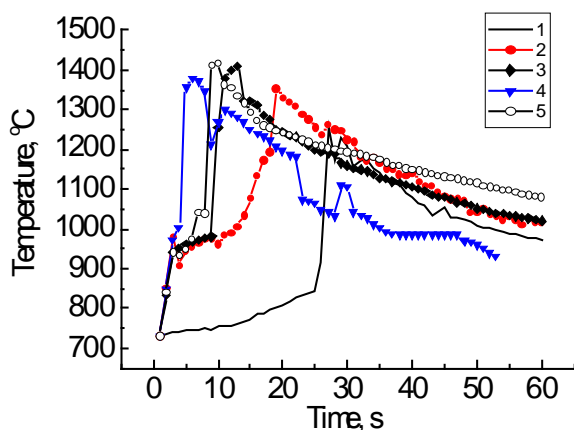
Combustion thermograms obtained with the help of a pyrometer have shown that in the course of

mechanochemical treatment of quartz, the modifying additives exert a significant effect on all combustion parameters: the induction period, combustion rate, maximum temperature of combustion and the change of heat conditions at the after burning stage (postprocesses). Fig. 1a, b present thermograms of combustion of the system  $\text{SiO}_2 + 37.5\% \text{ Al}$  with quartz in the initial state and modified with different additives. The presence of modifiers renders a considerable effect on the combustion process development decreasing the induction period and increasing the temperature of combustion.

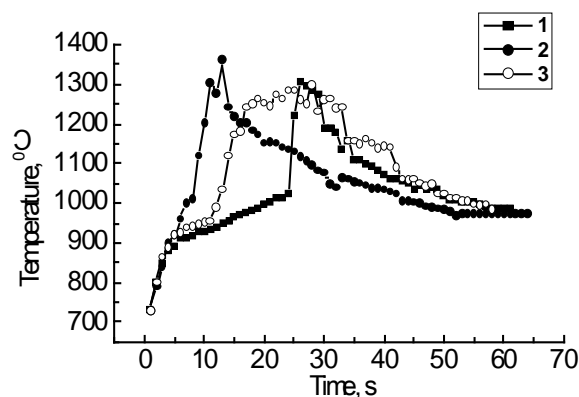
All the nitrogen containing additives used are characterized with splitting of ammonia molecule, when heated and then, ammonia displays reducing properties. So, it burns in the atmosphere of oxygen forming water and nitrogen.



Of the used nitrogen containing modifiers, the strongest effect on the change of combustion rate and temperature was exerted by carbamide which, when heated to  $150\text{--}160^\circ\text{C}$ , decomposes with formation of biuret –  $\text{H}_2\text{NCONHCONH}_2$ ,  $\text{NH}_3$  and  $\text{CO}_2$ , i.e. decomposition products contain ammonia and carbon dioxide. Then, oxidation of ammonia proceeds according to the above mentioned formula with formation of nitrogen and water which interact with aluminium. When heated to the melting temperature  $209^\circ\text{C}$ , dicyandiamide  $\text{HN}=(\text{NH}_2)\text{NHCN}$  decomposes with evolution of ammonia and formation of melanine ( $\text{HN}=\text{CNCN}$ ). aluminium resulting in the shift of the beginning of the mixture ignition, thereby increasing the combustion temperature.



a



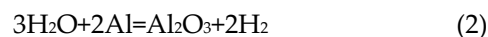
b

FIG. 1 THERMOGRAMS OF COMBUSTION OF QUARTZ WITH ALUMINUM: WITHOUT ADDITIVES (1) AND IN THE PRESENCE OF: A -  $\text{HN}=\text{C}(\text{NH}_2)\text{NHCN}$  (2),  $(\text{NH}_2)_2\text{CO}$  (3) AND  $\text{SiO}_2\text{-NH}_2\text{O}$  (4),  $(\text{C}_2\text{H}_5\text{OH})\text{N}$  (5); B-  $\text{C}_4\text{H}_6\text{O}_4$  (1),  $\text{NH}_4\text{OH}$  (2) AND MIXTURE  $\text{C}_4\text{H}_6\text{O}_4 + \text{NH}_4\text{OH}$  (3)

Of the used nitrogen containing modifiers, the strongest effect on the change of combustion rate and temperature was exerted by carbamide which, when heated to  $150\text{--}160^\circ\text{C}$ , decomposes with formation of biuret –  $\text{H}_2\text{NCONHCONH}_2$ ,  $\text{NH}_3$  and  $\text{CO}_2$ , i.e. decomposition products contain ammonia and carbon dioxide. Then, oxidation of ammonia proceeds according to the above mentioned formula with formation of nitrogen and water which interact with aluminium. When heated to the melting temperature  $209^\circ\text{C}$ , dicyandiamide  $\text{HN}=(\text{NH}_2)\text{NHCN}$  decomposes with evolution of ammonia and formation of melanine ( $\text{HN}=\text{CNCN}$ ).

When acids and alcohols were used as activating additives, it was stated that the process kinetics sharply increases: the induction period decreases, and the ignition rate grows resulting in the increase of combustion temperature. The greatest effect was observed in the presence of aqueous silicic acid and polyvinyl alcohol, i.e. compounds containing a great amount of bound water which, when heated (above  $200\text{--}400^\circ\text{C}$ ), separates and interacts with aluminium resulting in the shift of the beginning of the mixture ignition, thereby increasing the combustion temperature.

Thus, it may be concluded that bound water present in the mixture components makes a positive effect on the combustion process. The presence of water results in its interaction with aluminium, formation of hydrogen and aluminium oxide.



The reaction proceeds with evolution of a great amount of heat, and the formed hydrogen, a strong reducer, reacts with silicon oxide and reduces silicon according to the reaction.



To confirm this hypothesis, the systems containing succinic acid, aqueous solution of ammonia and the mixture of these two compounds as modifiers have been considered. The temperature curves of combustion show (Fig. 1b) that the presence of succinic acid decreases the induction period. Aqueous solutions of ammonia (ammonium hydroxide) enhance this effect increasing the temperature of combustion. Its participation in the combustion process is of a multi-stage character. Firstly, when heated, ammonium hydroxide molecule decomposes into water and ammonia. Then, the process of ammonia oxidation takes place with formation of water and nitrogen and now two molecules of water take part in the combustion process interacting with aluminium, and the formed hydrogen reduces silicon from quartz. In total, all these processes contribute to the decrease of the induction period and increase of the combustion temperature.

At the combined modification of quartz with succinic acid and ammonia solution, the processes get complicated. As it is known that under the action of ammonia on succinic acid there forms ammonium salt of succinic acid which, when heated, loses two molecules of water and transforms into a full amide of succinic acid. As further heated, amide of succinic acid loses one molecule of ammonia from its two aminogroups ( $-\text{NH}_2$ ) and transforms into a five-member cyclic compound—imide of succinic acid (imide group =  $\text{NH}$ ). Simultaneously, the process of silicon dioxide reduction goes. All the processes are reflected in the complex course of the temperature profile of combustion process showing the decrease in the induction period, though in a less extent than when only using ammonia solution, and higher temperature after the curve maximum. The multi-stageness of chemical reactions provides a higher level of temperature at the stage of postprocessors (the stage of afterburning) when the phase composition of synthesis products is formed.

Table 1 presents the results of measurements on the induction period of ignition, combustion rate and maximum temperature of the process depending on

the type of the modifiers used. Special attention should be paid to the role of bound water present in the modifying additives. The increase in its amount results in intensification of the combustion process due to oxidation of aluminium and formation of elemental hydrogen which acts as an active reducer of silicon dioxide.

TABLE 1 THE DEPENDENCY OF THERMOKINETIC CHARACTERISTICS OF THE COMBUSTION PROCESS (SHS) OF THE SYSTEM ( $\text{SiO}_2 + 37.5\% \text{ AL}$ ) ON THE TYPE OF MODIFIER AT MCT OF QUARTZ

Modifiers	Thermokinetic characteristics of the technological combustion process		
	The induction period, s	$V_{\text{comb}}(\text{up to } T_{\text{max}})$ , d/s	$T_{\text{max}}$ , °C
Without a modifier	25	16	1254
Dicyandiamide $\text{HN}=\text{C}(\text{NH}_3)\text{NHCN}$	7	32.5	1353
Carbamide – $(\text{NH}_2)_2\text{CO}$	7	100	1400
Ammonium dioxide - $\text{NH}_4\text{OH}$	5	130	1355
Succinic acid - $\text{C}_4\text{H}_6\text{O}_4$	20	60	1300
Succinic acid+ammonium dioxide - ( $\text{C}_4\text{H}_6\text{O}_4 + \text{NH}_4\text{OH}$ )	12	32.7	1285
Water containing silicic acid - $\text{SiO}_2 \cdot n \text{H}_2\text{O}$	3	96	1379
Polyvinyl alcohol - $(\text{C}_2\text{H}_5\text{OH})_n$	5	100	1416

Thus, using mechanochemical activation and modification of the surface layers of the particle dispersed, it is possible to purposefully effect the development of combustion process, its thermodynamic characteristics, which must be finally realized in the phase composition and properties of the material synthesized.

Activation of the combustion process using different additives-modifiers is related directly to both the presence of a concrete chemical compound in the charge mixture and the change in the structure and state of the surface of quartz particles as a result of their grinding in the presence of additives. Fig. 2 presents electron-microscopic pictures of quartz particles in the initial state and after modification with additives providing the greatest activation of the combustion process of the system under study.

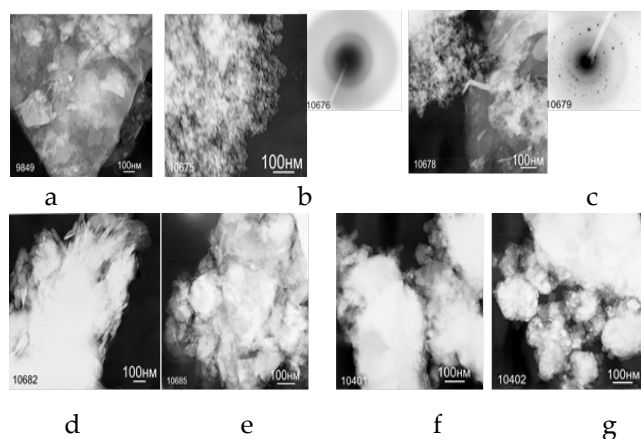


FIG. 2 THE MICROSTRUCTURE OF QUARTZ PARTICLES IN THE INITIAL STATE (A) AND AFTER GRINDING WITH SILICIC ACID (B,C), POLYVINYL ALCOHOL (D,E) AND CARBAMIDE (F,G)

Quartz used in the work is distinguished by the presence of a sufficiently great amount of impurities (up to 18.7% of microcline –  $K(Si_3Al)O_8$ ) and laminated structure of the particle surface (Fig. 2a). It imparts a specific coloration to the particles due to natural modification of their surface layer.

When aqueous silicic acid is introduced into the quartz powder, a highly dispersed amorphous film is formed on the surface of particles (Fig. 2 b,c). The presence of two kinds of particles in the powder was detected that do not reveal structural degree of order which are likely to be formed by silicic acid (visible size 10-20 nm) and smaller crystalline particles (granular formations) with most probable cubic structures. The rounded particles of the first kind occur more frequently both in the form of independent formations and attached to quartz particles.

When milling with polyvinyl alcohol, partial destruction of the surface layer of quartz particles and formation of a structured film on the particle surface take place (Fig. 2 d,e). At MCT of quartz with carbamide additives, on the surface of particles dispersed, a film with a loose structure and a great amount of nanodisperse formations along the boundary of particles form. The particles may transform under the action of electron beam of the microscope. Apart from the change of the particle morphology, one can observe the appearance of “active” particles which can interact with amorphous hydrogen of the preparation support.

Of special interest is the results on the change in the morphology of quartz particles in the course of their

modification with aqueous solution of ammonia, succinic acid and their mixture. (Fig. 3). In the first case, a dense structured film on the surface of quartz particles is observed. In the presence of succinic acid, the process of destruction of particles intensifies. After treatment in the mixture of ammonium dioxide and succinic acid, on the one hand, dispersibility of quartz intensifies, on the other hand— ultrafine particles form conglomerates of different volume and configuration. Such formations are characterized with high activity and transform under the effect of electron beam in the microscope.

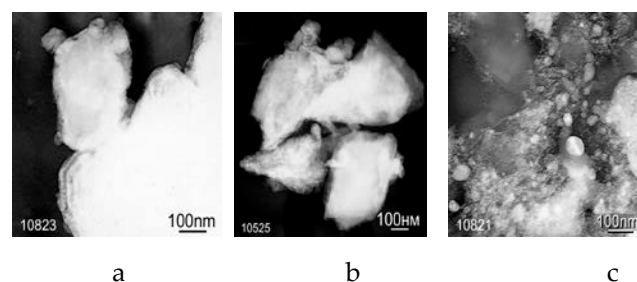


FIG. 3 ELECTRON-MICROSCOPIC PICTURES OF QUARTZ AFTER MCT IN THE PRESENCE OF –  $NH_4OH$  – AMMONIUM DIOXIDE (A),  $C_4H_6O_4$  – SUCCINIC ACID (B) AND THE MIXTURE OF SUCCINIC ACID+AMMONIUM DIOXIDE –  $C_4H_6O_4 + NH_4OH$  (C)

Such morphological peculiarities of the particles of the mineral under study after MCT in the presence of the mixture ( $C_4H_6O_4 + NH_4OH$ ) are conditioned by the interaction of succinic acid and ammonia solution in the process of MCT first with separation of bound water, then ammonia and formation of a cyclic compound—imide of succinic acid. Water and ammonia formed contribute to destruction of the crystalline structure of quartz, accelerate the process of dispersion and formation of nanodisperse particles, resulting in hydration of the surface and agglomeration of particles. A highly active surface layer interacts also with ammonia and imide of succinic acid resulting in formation of film structures encapsulating the particle of mineral.

The specific character of the particle surface structure counts for, as shown above, the thermokinetic peculiarities of the combustion process of such material. Kinetic and thermal characteristics of combustion determine the final result of combustion process—the phase composition of synthesis products on which the exploitation properties of the material depend. Table 2 presents the results of X-ray phase analysis of synthesized samples on the basis of quartz with participation of additives-modifiers.

TABLE 2 THE PHASE COMPOSITION IN THE COMBUSTION PRODUCTS OF SAMPLES  $\text{SiO}_2 + 37.5\% \text{ Al}$  DEPENDING ON THE TYPE OF MODIFIER AT MCT OF QUARTZ

Modifiers	Phase content, %					
	Phases					
	$\text{Al}_2\text{O}_3$	Si	$\text{AlN}$	$\text{FeAl}_3\text{Si}_2$	$\text{SiO}_2$	Al
Without a modifier	48.4	12.9	-	1.8	24.7	12.2
$\text{HN}=\text{C}(\text{N}_2)\text{NHCN}$	59.9	15.4	4.1	1.4	10.7	9.5
$(\text{NH}_2)_2\text{CO}$	62.2	20.5	6.2	1.8	7.0	2.3
$\text{NH}_4\text{OH}$	53.1	15.4	4.8	1.8	16.4	8.5
$\text{C}_4\text{H}_6\text{O}_4$	43.6	14.9	-	1.4	29.9	10.2
$\text{C}_4\text{H}_6\text{O}_4 + \text{NH}_4\text{OH}$	54.5	24.5	9.5	1.3	7.7	2.5
$\text{SiO}_2 \cdot n\text{H}_2\text{O}$	61.7	14.0	2.8	5.5	10.5	5.6
$(\text{C}_2\text{H}_3\text{OH})_n$	60.2	16.3	7.8	2.6	11.1	3.0

First of all, it follows the presented data that modification of quartz with different additives in the course of MCT contributes to a more complete development of redox processes with formation of the main phase—corundum, reduced silicon and formation of aluminium nitride. The most complete realization of initial aluminium and reduction of silicon take place in the presence of carbamide and the mixture of succinic acid with ammonium dioxide in the charge. Aluminium nitride is formed due to interaction of aluminium with nitrogen of air and nitrogen present in the modifying additives. The amount of aluminium nitride in the reaction products rises with the increment of the combustion temperature. The presence of a sufficient amount of bound water and ammonia group in the modifying additives positively effect the increment in the amount of the matrix phase (corundum) and aluminium nitride. Of special attention is the results on the role of bound water present in the modifying additives. The increase in its amount affects the yield of corundum in synthesis products that in combination with the presence of aluminium nitride must count for the increase in the quality of the material regarding its strength and heat stability.

## Conclusion

Thus, a positive effect of modification of the charge components with different additives has been demonstrated which activate the combustion process of the systems based on silicon dioxide. The presence of a sufficient amount of bound water and ammonia group in additives—modifiers contributes to the

increase in the amount of the matrix phase (corundum) and aluminium nitride in synthesis products. Specific peculiarities of the effects of different additives on the combustion process, namely, the increase of kinetic characteristic and combustion temperature of the system, are stated. It is stated that the most complete realization of initial aluminium and reduction of silicon take place in the presence of carbamide and the mixture of succinic acid with ammonium dioxide in the charge.

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